Fe 2p absorption in magnetic oxides: quantifying angular dependent saturation effects

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INTRODUCTION

Saturation effects related to indirect detection of x-ray absorption spectra, like total electron yield (TEY) or fluorescence yield measurements, are of interest for applications of linear and circular dichroism (respectively XMLD and XMCD). XMCD studies on Fe, Co and Ni using the L-edge TEY spectra have shown that saturation can cause errors in excess of 100% on the extracted magnetic moment values [1]. In TEY measurements, the intensity is given by

$$I(\alpha, E) = \frac{A\,d}{d + \lambda(E)\sin\alpha} \qquad (1)$$

where A is the number of electrons produced per photon, α is the angle of incidence, *d* is the probing depth, and λ(E) is the absorption length. The relevant parameter for angle-dependent saturation effects is the ratio between *d* and λ(E)sinα, i.e. the effective absorption length projected along the surface normal. For a given α and for $d << \lambda(E)\sin\alpha$, the measured yield is inverse proportional to the absorption length, i.e. proportional to the absorption coefficient μ

$$I(\alpha, E) \approx \frac{A\,d}{\lambda(E)\sin\alpha} \propto \frac{1}{\lambda(E)} = \mu(E) \qquad (2)$$

If *d* cannot be neglected with respect to λ(E)sinα this direct proportionality between I and μ is lost and angular dependent saturation occurs. As both quantities change from pure metals to oxides, saturation effects are expected to be, for a given element and core excitation, different for metallic or oxide compounds. However, a clear trend for different materials (oxides vs. metals) has not been identified yet. Previous works have estimated saturation effects for metals at absorption resonances having large cross sections, like the $L_{2,3}$ edges of transition metals [1,2] and the $M_{4,5}$ edges of rare earths [3]. Resulting *d* values were found to be strongly material-dependent and much shorter than expected. In contrast, quantitative studies concerning saturation effects in insulating materials and oxides are scarce. Magnetic oxides are currently the focus of considerable interest because of their applications in spin electronics [4]. The potential of XMLD and XMCD for studying their magnetic properties has been recently shown [5] and the impact of saturation effects needs to be considered also for this class of materials.

RESULTS

The samples were thin Fe_3O_4 layers epitaxially grown on α-Al_2O_3 (0001) single crystals by atomic oxygen assisted MBE [6]. The thickness of the Fe_3O_4 layers ranged from 5 to 240 Å. The experiments were performed at the Advanced Light Source (Berkeley), using beamline 6.3.2 [7]. Fig. 1 shows the maximum intensity, multiplied by sin(α), at the L_3 and the L_2 resonances for an Fe_3O_4 layer 240 Å thick. In absence of saturation, I(α,E)sin(α) should be independent of α. On the contrary, we observe a strong reduction when the angle of incidence decreases. For a given α, the intensity is more severely reduced at the L_3 than at the L_2 edge. The two experimental curves are fitted with the expression of eq.(1) using A and λ/d as free parameters. We find that the best fits correspond to λ/d = 3.5 at the L_3 edge and to λ/d = 9 at the L_2 edge. *d* was also estimated experimentally from the thickness dependent values of the L_3 intensity normalized to the

background before the resonance. Fig. 2 shows the normalized peak height at the L_3 edge plotted as a function of the Fe_3O_4 layer thickness. The curve fitting to an exponential function y = A[1-exp(-x/d)] gives d = 45 Å, which is roughly three times the value estimated for pure metallic Fe [1]. λ(E) was derived by inversion of the absolute absorption intensity, obtained by scaling the experimental $L_{2,3}$ TEY spectrum measured at $\alpha = 90°$ to the calculated atomic photoabsorption cross section. The λ(E) values obtained at photon energies corresponding to the maximum of the 2p edges are $\lambda(L_3)$ = 170 Å and $\lambda(L_2)$ = 525 Å.

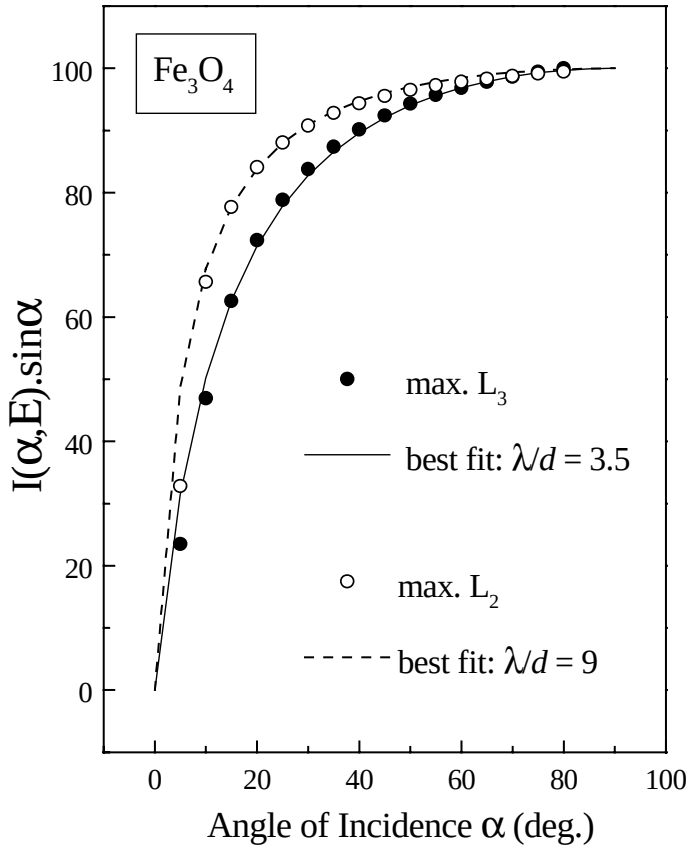

Figure 1 I(α,E)sinα as a function of α for an Fe_3O_4 layer of 240 Å. Solid and open circles correspond to the maximum of the L_3 and the L_2 edges, respectively. The best fits correspond to λ/d = 3.5 for the L_3 edge (solid line) and to λ/d = 9 for the L_2 edge (dashed line).

Our study evidences one general aspect of TEY saturation, i.e. its material dependence. It is interesting, for instance, to compare our results for Fe_3O_4 with those reported for metallic Fe. For Fe, the values reported in the literature are $\lambda(L_3)/d$=10 and $\lambda(L_2)/d$=19 [1]. In the oxide, the reduced density of Fe ions tends to increase λ, hence to weaken saturation, but their reduced 3d occupancy induces stronger oscillator strengths, and acts in the opposite direction. Moreover, we have found a TEY probing depth for the oxide about three times larger than for the metal. The net result is, for a given angle, a stronger saturation for the oxide than for the metal. As a consequence, quantitative magnetic studies based on linear and circular dichroism at the 2p edges of Fe in magnetic oxides must take into account and correct for saturation effects.

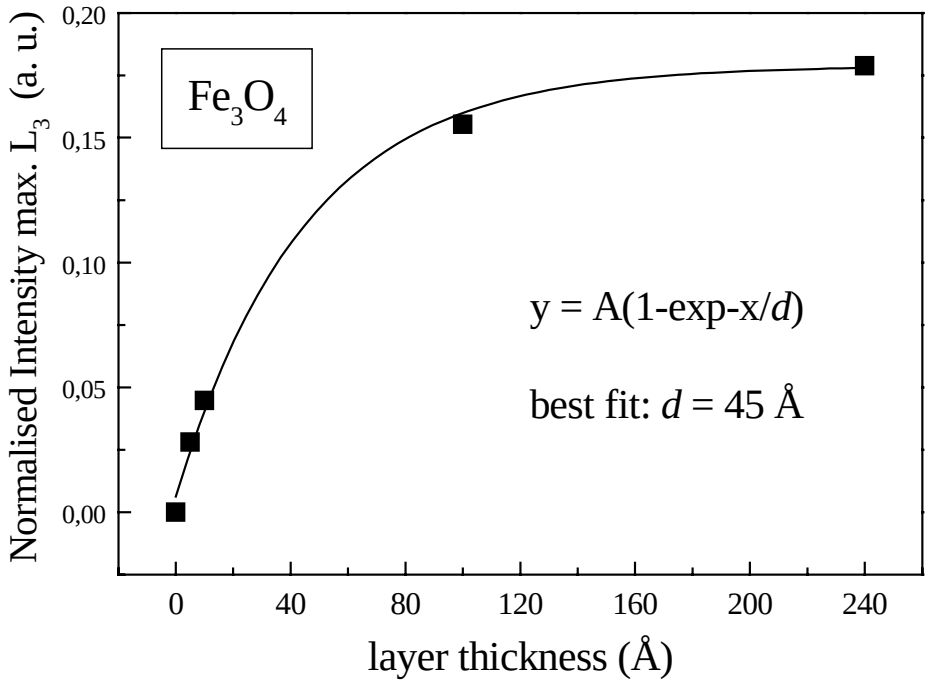

Figure 2 Normalized peak height at the Fe-L_3 edge plotted as a function of the Fe_3O_4 layer thickness. The best fitting curve, using the probing depth as a free parameter, corresponds to d = 45 Å.

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